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Study of synthesized molecular chiral lactam structures for volatile compound profile enrichment using gas chromatography–mass spectrometry (GC-MS) technique

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INTRODUCTION & AIM

In a study of the chiral enantiomer structure of cyclic trilactams possessing C3 symmetry, the dimer structure was established through the formation of three NH···O type complementary H-bonds at three amides which were applied as agents for volatile compound profile extraction in a sample via the liquid phase extraction technique.

The extraction efficiency of cyclic trilactam materials for analytes found in perfume samples was assessed using HS-SPME-GC-MS. The experimental arrangement of perfume solution was combined with the materials in EtOH solvent in an open vial, while the perfume in EtOH (without the materials) served as the control. Following overnight evaporation at room temperature, the remaining liquid was collected for HS-SPME-GC-MS analysis. Extraction took place overnight to explore the materials' extraction efficiency and facilitate re-crystallization. The materials underwent re-crystallization, with the collected crystals revealing a similar shape to the observed dimers. This suggests a robust hydrogen bonding interaction within the dimer and the volatile compounds interacting. A list of identified compounds during extraction was compiled for each material and the control. The enrichment peak areas in the chromatogram results were determined as the analyte peak area in the sample containing the materials to the area of the same analyte in the control. This indicated the extraction performance of volatile analytes in the sample containing the materials compared with the control sample, such as Linalylformate $(5.1\pm0.015)\times10^7$, Citronellol $(5.1\pm4.2)\times10^6$, Carvone $(4.2\pm3.5)\times10^5$, Linalylacetate $(2.5\pm2.2)\times10^7$ and α-Terpinylacetate $(2.3\pm1.7)\times10^5$ of the main potential compounds. Higher enrichment signifies stronger interactions between the analytes and these materials.

The solubility between the compounds in less polar solvents can be attributed to their structural features and the nature of the solvent. Specifically, the solubility of each compound is influenced by factors such as polarity, hydrogen bonding capability, and the molecular size and shape of the compounds can influence their interactions with the solvent molecules, further impacting solubility differences between compounds in less polar solvents.

METHOD

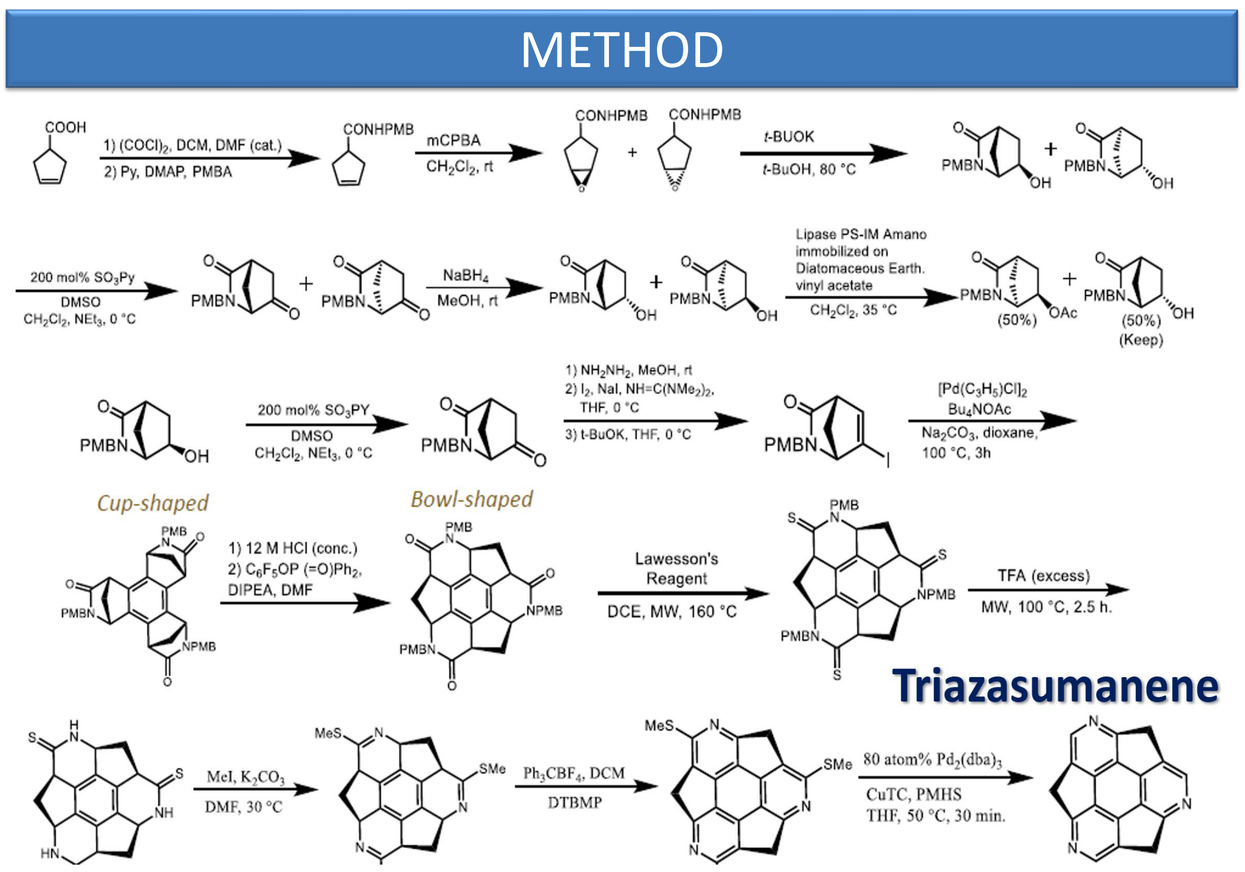


Figure 1. The synthesized mechanisms of cup-shaped cyclic trilactams

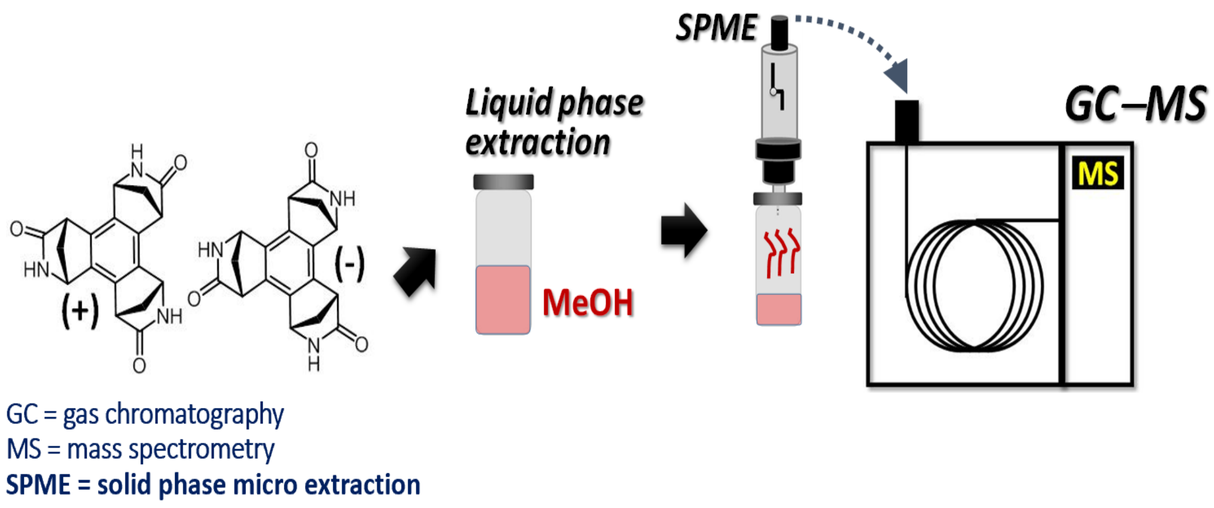
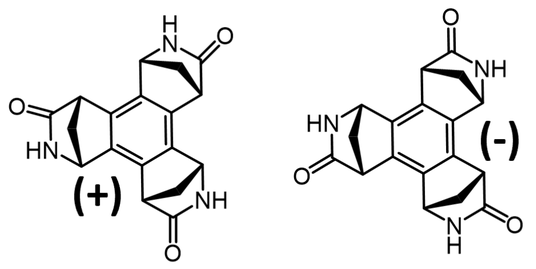


Figure 2. Extraction of different compounds in perfume

RESULTS & DISCUSSION



Cup-shaped trilactam

Avg. N(H)-O 2.86 Å

Solid form showed dimer structure

Prepared by vapor diffusion with $CHCl_3$/Hexane

Space group : $P2_1$
a = 9.6912(4) b = 16.4626(4)
c = 11.3683(3)
α = 90 β = 103.690(3) γ = 90
Cell volume = 1762.2
R-factor (%) = 5.88

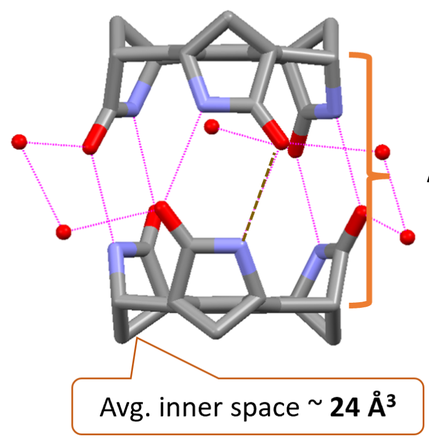


Figure 3. Molecular chiral structure of *(+)*-1 and *(−)* of cup-shaped trilactams

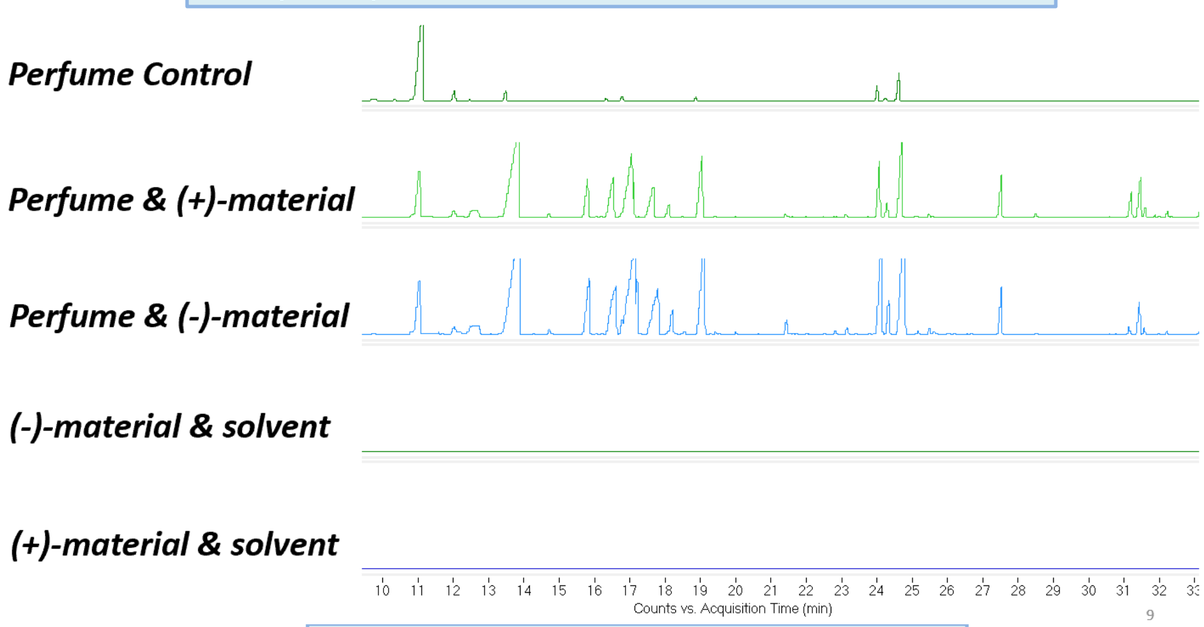


Figure 4. GC-MS of perfume extracts

Compound Name	*I* lit	Peak area in		
		control	Perfume-(-)-1	Perfume-(+)-1
Linalyl formate	1215	0 ± 0	$(5.1\pm0.015)\times10^7$	$(6.1 \pm 1.8) \times 10^7$
Citronellol	1228	$(1.1 \pm 2.6) \times 10^5$	$(5.1 \pm 4.2) \times 10^6$	$(6.2 \pm 2.2) \times 10^6$
Carvone	1242	$(0.10 \pm 0.10) \times 10^4$	$(4.2 \pm 3.5)\times10^5$	$(7.3 \pm 2.7) \times 10^5$
Linalylacetate	1257	$(5.5 \pm 3.5) \times 10^6$	$(2.5 \pm 2.2) \times 10^7$	$(4.2 \pm 1.2) \times 10^7$
α-Terpinylacetate	1350	$(3.4 \pm 3.2) \times 10^4$	$(2.3 \pm 1.7)\times10^5$	$(4.1 \pm 2.2) \times 10^5$

Table 1. Compound profiles, including retention indices from literature (*I*lit), peak area (counts/second) with were analyzed using GC-MS. The analysis identified potential compounds that exhibit significant chiral recognition.

CONCLUSION

In this study, the effectiveness of the synthesized cup-shaped trilactams was evaluated through their ability to extract chiral compounds from perfume. The materials demonstrated strong enrichment performance for these compounds. The examined selective interaction mechanisms expand the potential uses of these materials, including applications in host–guest chemistry and chiral recognition.

FUTURE WORK / REFERENCES

[1] S. Sartyoungkul, I. Thaveesangsakulthai, MKE. Cabello, C. Kulsing, H. Sakurai. Application of cup-shaped trilactams for selective extraction of volatile compounds by gas chromatography-mass spectrometry. Analyst. 2020, 145, 6668-6676.

[2] Q. Tan, P. Kaewmati, S. Higashibayashi, M. Kawano, Y. Yakiyama, H. Sakurai. Triazasumanene: An Isoelectronic Heteroanalogue of Sumanene. Bulletin of the Chemical Society of Japan, 2018, 91, 531-537.